

MORPHOLOGY ANALYSIS AND CORROSION PERFORMANCE ON ALUMINUM ZIRCONIUM COMPOSITE

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ABSTRACT

The high strength aluminum alloy 5083 of composition 0.4Si 0.4Fe 0.1Cu 0.4Mn 4Mg 0.25 Zn 0.15Ti 0.05Cr and Al balance which has high yield strength of around 200 MPa and tensile strength of 300 MPa and good corrosion resistance for sea water and saline environments, is alloyed with zirconium (Zr), which is highly resistant to heat and corrosion, very strong, malleable and ductile, at various compositions. The alloying element zirconium metal powder is mixed at different proportions of 0.3 wt%, 0.5 wt% and 0.7 wt% with aluminum 5083 as the base metal. Corrosion resistance and morphological properties of the samples were investigated by potentiostatic polarization, scanning electron microscope (SEM) and x-ray diffraction (XRD) tests. The corrosion study has been carried out in 3.5 % NaCl solution at a sweep rate of 1mV/s. After open circuit potential has become stable, the pitting corrosion behavior has been studied after 6 hours of immersion, at a temperature of 350C and a pH value of 7. The results show that the addition of zirconium has increased the corrosion resistance of the aluminum alloy, 0.7 wt% of zirconium sample has given the best results and great resistance against corrosion, allowing them to be applicable for direct application for saline environment.

KEYWORDS: Aluminium-Zirconium Composite, Aluminium, Zirconium Composite, Nanocomposites, Aluminium Composites, Corrosion Performance & Morphology Analysis

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INTRODUCTION

When aluminium is mixed with zirconium it will increase the fracture toughness [1]. In view of this, Al 5083 as matrix and reinforcing it with zirconium particles result in MMC's with high specific strength for various light weight applications. Aerospace and automobile industries uses widely Al – based MMC's for its various components and also AL – based MMC's are used in architectural applications. Over the last three decades, particles reinforced MMC's have been the most popular and among the MMC's, reinforced Al-MMC's are finding wide applications. Incorporations of the second phase particulate into an aluminium matrix material will enhance the mechanical properties of the base aluminium matrix material: it will significantly change the corrosion properties and its behavior.

The physical and mechanical characteristics of the nano-sized zirconium particles make them suitable for being used as reinforcement in aluminium based MMC's. Zirconium nano powder of various composite composition weight percentages and the corrosion study is mainly observed in this study, using polarization and electrochemical impedance studies corrosion properties were evaluated. The studies show that increasing the

weight percentage of zirconium has improved the corrosion resistance properties of the Aluminium of the grade 5083 and also the mechanical properties are also improved according to the detailed study. The hardness properties were also improved of the base material and has shown some real improvement and many more characteristically development in the base material.

Table 1: Properties of Metals Incorporated

Properties	Unit	Aluminum	Zirconium
Density	g/cm ³	2.7	6.5
Tensile strength	MPa	290-390	211-1400
Hardness	Hv	2200	1200
Young modulus	GPa	68	200
Fracture toughness	MPa.m ^{1/2}	4.40	1.54-4.07
Melting Point	°C	654	2700

In summary, the work presented in this project can be classified into the following points.

- To study and compare the results of the aluminum and zirconium made by the conventional metal casting process with varying weight percentages of zirconium in the MMC's.
- To investigate the corrosion properties and study the surface and crystalline morphology of aluminum- zirconium composites.

EXPERIMENTAL

For the preparation of the composites, high purity aluminium of grade 5083 and high purity zirconium nano powder of size 50 nm were used in the project. The aluminium was melted in the muffle furnace at a temperature of 1000°C and also zirconium nano powder was also separately preheated at the same temperature in the muffle furnace, followed by stirring, degassing with inert gas (Ar)[1-3]. The zirconium was added to the composite at varying compositions of weight percentages explained in the given table.

Table 2: Chemical Composition of Studied Composites (in wt %)

Alloy	Zr	Fe	Si	Alloy
Al-0.3Zr	0.3	0.1	0.4	Balance
Al-0.5Zr	0.5	0.1	0.4	Balance
Al-0.7Zr	0.7	0.1	0.4	Balance

The cast ingots had a mass of 350g each and cooled at room temperature for about 2 hours and were sectioned into different dimensions for each test and surface finished. The cutting were achieved by wire cut electrical discharge machining process (Wire cut EDM process).

The tests performed on the specimens are scanning electron microscopy, x-ray diffraction and electrochemical testing.

Table 3: Specimen Dimension for Each Test (in mm)

Test	Specimen Dimensions
SEM	10*10*10
XRD	30*30*3
ECT	20*20*3

Scanning Electron Microscopy Test

The scanning electron microscopy test was performed on the polished specimen after the casting of the composites and cooling at the room temperature. The results of SEM tests include the imaging of specimen at different focal lengths, say 10 μm and 50 μm by JEOL JSM-6460 scanning electron microscope.

X-Ray Diffraction Test

The x-ray diffraction test was performed to find out the purity and the nobility of the composite and the bulk composition of the composite fabricated.[20-26] The conditions of the test were : Drive axis: Theta- 2Theta, Scan range: 5.0000 - 80.0000 deg, Scan mode: Continuous Scan, Scan speed: 10.0000 deg/min, Sampling pitch: 0.1000 deg, Preset time: 0.60 sec, Smoothing: AUTO, Smoothing points: 5, B. G. Subtraction: AUTO, Sampling points: 5, Repeat times: 30, Ka1-a2 Separate: MANUAL, Ka1 a2 ratio: 50%, Differential points: 5, FWHM threshold: 0.050 deg, Intensity threshold: 30 par mil.

Electrochemical Test

In a electrode glass beaker of 500 ml electrolyte, the potentiodynamic polarization and linear polarization study was done. A saturated calomel electrode (SCE) and two graphite electrodes are taken as reference and auxiliary electrodes. The electrode (aluminium or AA5083-Zr composite) is fixed in a teflon holder in which 2 cm² of surface was exposed to 3.5% NaCl solution. It is finished with 1200 metallographic finish and cleaned with distilled water[27-30]. The electrochemical measurements were carried out in the test solution after reaching the open-circuit potential (E_{ocp}).

Potentiodynamic polarization were done at scan rate of 1.0 mV s⁻¹ in the potential range from ± 150 mV with respect to the E_{ocp} , linear polarization measurements were done in the potential range ± 20 mV from the corrosion potential with scanning rate of 0.50 mV s⁻¹. [4-9]. The polarization curves obtained are completely linear in this potential domain, the polarization resistance, R_p , was determined from the data collected in potential range ± 5 mV from the corrosion potential.

After immersion of the samples for about 6 hours as described above in electrochemical testing the pitting corrosion test has been studied, to understand the corrosion format in the specimens provided. The chemical composition (wt %) of pure aluminium used in this study is 0.3Zr, 0.4Si, 0.4Fe, 0.1Cu, 0.4Mn, 4Mg, 0.25Zn, 0.15Ti, 0.05Cr and Al balance; Specimen 2 has 0.5Zr, 0.4Si, 0.4Fe, 0.1Cu, 0.4Mn, 4Mg, 0.25 Zn, 0.15Ti, 0.05Cr and Al balance: Specimen 3 has 0.7Zr, 0.4Si, 0.4Fe, 0.1Cu, 0.4Mn, 4Mg, 0.25 Zn, 0.15Ti, 0.05Cr and Al balance while the composition of the AA5083 alloy sample is 0.4Si, 0.4Fe, 0.1Cu, 0.4Mn, 4Mg, 0.25 Zn, 0.15Ti, 0.05Cr and Al balance.

RESULTS AND DISCUSSIONS

Polarization Measurement

The inspection of the figure 1 shows that with increase or addition of the zirconium nano powder in the aluminium 5083 metal, there has been a considerable increase in the corrosion current densities, which can directly related to the corrosion resistance of the metal and the polarization resistance. As said this test has been carried out at 3.5 % NaCl solution that could be the mean ocean water condition. The above test results justify that the increase of the zirconium weight percentage in the composite there has been a considerable reduction in corrosion rate and a proper improvement in

the corrosion resistance [10-15].

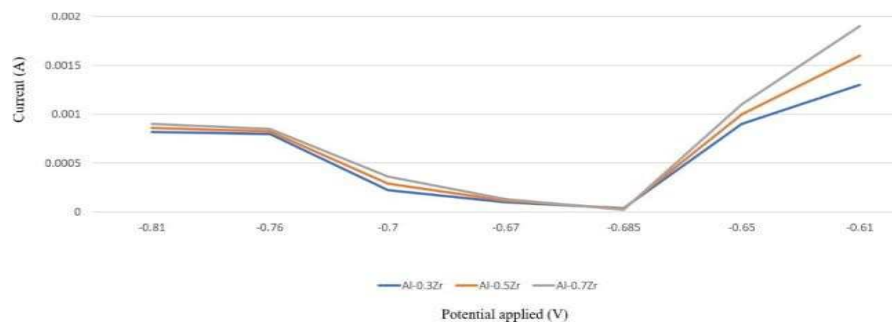


Figure 1: Potentiodynamic Curves Obtained for a) Al-0.3Zr b) Al-0.5Zr and c) Al-0.7Zr in 3.5 % NaCl Solution

According to E_{corr} values obtained during the test exhibited in table 4 it shows that from the alloy composition Al-0.3Zr to Al-0.5Zr there has been a certain decline in the voltage observed in the reading and on the other hand the values of i_{corr} having a considerable uphill, giving away that the resistance which is the potential resistance has been improved throughout the study. And the 3.5 % NaCl solution has dedicated a lower part on the shifting of the noble aluminium, with reference to its blank alloy [27-30]. Also the increasing of the zirconium can be clearly be concluded from the entire study is that, the zirconium atoms have a great binding power and improving the corrosion resistance of the base metal aluminium and can be better be applicable for the marine applications.

The other data like the polarization resistance, current densities and corrosion rate are clearly tabulated in the table 5 and the unit of measurements are also mentioned along with the data.

Table 4: Potentiodynamic Polarization Parameters Obtained for Aluminium Alloys a) Al-0.3Zr b) Al-0.5Zr and c) Al-0.7Zr in 3.5 % NaCl solution

Specimen	ba (V/dec)	bc (V/dec)	E_{corr} , Calc (V)	E_{corr} , Obs (V)
Al-0.3Zr	0.02470	0.03486	-0.6850	-0.6852
Al-0.5Zr	0.05816	0.03940	-0.6855	-0.6856
Al-0.7Zr	0.06597	0.04332	-0.6859	-0.6858

According to the corrosion current density values given in Table 4, it is evident that Al-0.7Zr is being observed for more corrosion voltage in 3.5 % NaCl than Al-0.3Zr alloy (0.6858 V and 0.6852 V respectively).

The data given in Table 5 show that Al-0.7Zr is more resistant than other alloys i.e., Al-0.5Zr and Al-0.3Zr since the R_p value for Al-0.7Zr is higher (58.18 Ω) than that obtained for Al-0.5Zr (43.772 Ω) and Al-0.3Zr (29.426 Ω). This observation confirms that these results are in good agreement with those obtaining from the Tafel extrapolation method.

Table 5: Corrosion Parameters Obtained for Aluminium Alloys a) Al-0.3Zr b) Al-0.5Zr and c) Al-0.7Zr in 3.5 % NaCl Solution

Specimen	Corrosion rate (mm/year)	R_p (Ω)	i_{corr} (A)	j_{corr} (A/cm ²)
Al-0.3Zr	6.9621	29.426	0.00059	0.00059
Al-0.5Zr	4.6164	43.772	0.00039	0.00039

Al-0.7Zr	2.2707	58.1 18	0.00019	0.00019
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The values of R_p constantly increase with increasing zirconium concentration, while the current density values decrease. The polarization resistance value is the highest at weight percentages of 0.7 zirconium, while higher R_p value indicates lower corrosion rate. Therefore, in agreement with the Tafel extrapolation method, increasing the zirconium composition in the alloy the polarization resistance increases, indicating a lower corrosion rate. Hence addition of zirconium to aluminium 5083 has improved the corrosion resistance to the base metal.

SEM Analysis

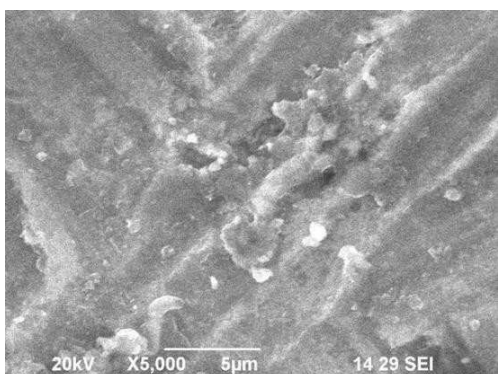


Figure 2: SEM Image of Al-0.3Zr

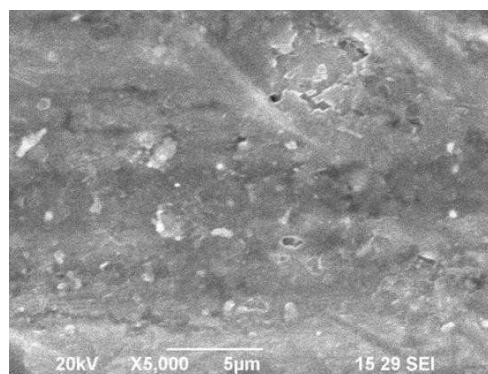


Figure 3: SEM Image of Al-0.5Zr

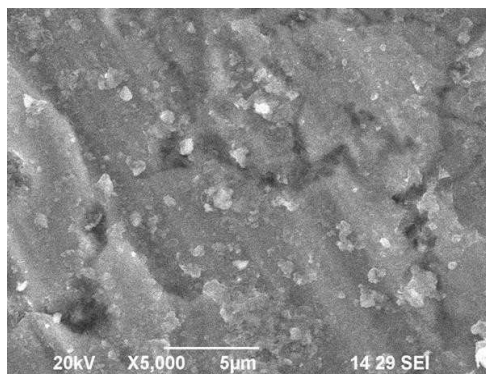


Figure 4: SEM Image of Al-0.7Zr

The above figures 2, 3 and 4 are the results of the sem analysis of the aluminium alloys after casting. The figure 2 exhibits the alloy compounding Al-0.3Zr and has the lowest composition of the zirconium nano powder in the alloy comparatively. And an increasing quantity of zirconium quantity has been observed in the figure 3, exhibiting the alloy Al- 0.5Zr and even more quantity in figure 4, exhibiting the alloy Al-0.7Zr. All the images show that they have uniform mixture of the zirconium nano powder in them.

In the sem images, the white spots are the zirconium nano powder in the alloy, while the black spots are the porosity in the alloy, which mainly been spotted in the specimen 2 which has Al-0.5Zr. This might be due to a slower stirrer speed or impurities in the casting process. While the rest of the portion is the microstructure is representing the aluminium 5083 which is the base metal. All the sem images are of X5000 by the JEOL JSM-6460 scanning electron microscope.

XRD Analysis

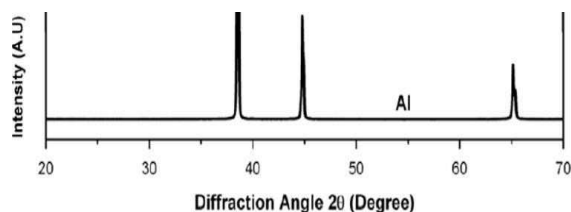
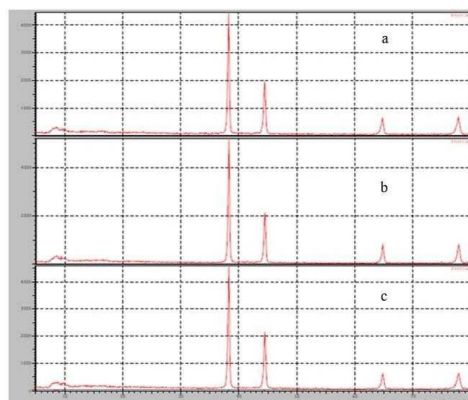


Figure 5: XRD Results of Pure Aluminium 5083



**Figure 6: XRD Results of Fabricated Alloys
a) Al-0.3Zr b) Al-0.5Zr and c) Al-0.7Zr**

X- ray diffraction (XRD) analysis was carried out for aluminium alloys Al-0.3Zr, Al-0.5Zr and Al-0.7Zr. The composite fabricated was of range 2θ is ranged between 00 and 800, and the comparison was made with pure aluminium 5083. It is clear that from the figure 4 aluminium has 3 peaks alone at $2\theta = 380, 450$ and 660 . And the intensity was also considerably low, around 3000. But on addition of zirconium the number of strong peaks has been raised to 6 to 8 peaks, bringing in new peaks at 50, 80, 370 and 780 on a whole for the three specimens. But the intensity of the values has been increasing from 6273, 6595 and 6954, showing the increasing intensity of zirconium nano powder in the alloy.

The table 6 gives the values of the three strongest peaks of the alloys a) Al-0.3Zr, b) Al-0.5Zr and Al-0.7Zr. And also the 2θ values of the peaks including with the d values and intensity counts. The strongest peak of all the specimen is found around $2\theta = 380$, with increasing values of 6273 for Al-0.3Zr, For Al- 0.5Zr the intensity count is 6595 and finally for Al- 0.7Zr the value obtained was 6954 counts. This increase in the intensity shows the increasing the weight percentages of the zirconium nano powder.

**Table 6: Strongest 3 Peaks of the Aluminium Alloys
Al-0.3Zr b) Al-0.5Zr and c) Al-0.7Zr**

Specimen	Peak no.	2Theta (deg)	d (Å)	Intensity (Counts)
Al-0.3Zr	3	38.2459	2.35137	6273
	5	44.4603	2.03606	3454
	8	77.8021	1.22664	1262
Al-0.5Zr	3	38.2496	2.35115	6595
	4	44.4553	2.03628	3615
	6	77.8260	1.22630	1645
Al-0.7Zr	5	38.2411	2.35165	6954
	6	44.4505	2.13649	3782
	8	77.7717	1.22704	1842

From the above results it can be concluded that the crystalline structure of the zirconium nano powder has improved the density and the structure of the aluminium 5083. And also the porosity of the aluminium 5083 has been reduced due to the addition of the zirconium nano powder.

From bragg's equation [13-16] we can find out the crystalline structure of the zirconium in the alloy. The equation is given below.

$$d = \frac{a}{\sqrt{h^2+k^2+l^2}}$$

The above equation shows the lattice spacing of the crystalline structure. h, k and l are the miller indices of bragg plane [13-16]. By calculating the lattice space from the above equation is calculated and was found out to be zirconium nano powder. And the increasing intensity value gives out the increasing weight percentages of the zirconium nano powder in the alloy.

CONCLUSIONS

The aim of this thesis was to study the morphology and corrosion performance of the aluminum and zirconium composite, with varying weight percentages of zirconium added to the base metal aluminum, to improve its efficiency for marine applications and bone implants. The corrosion performance, which has been mainly studied in this project, the conclusions which have been drawn from this investigation are: Addition of zirconium had improved the corrosion resistance ability of the base metal i.e., aluminum (from 8Ω to 58Ω). The corrosion performance has been improved by the addition of the zirconium nano- powder to aluminum. The corrosion resistance of the 3 specimens in descending order are: 0.7Zr < 0.5Zr < 0.3Zr which clearly denotes that increasing the weight percentage of the Zirconium has improved the corrosion resistance of aluminum of grade 5083. The corrosion rate of the different weight percentages of zirconium (0.3wt%, 0.5wt% and 0.7wt%) can be concluded to be 0.7Zr = 2.2707 mm/year, 0.5Zr = 4.6164mm/year and 0.3Zr = 6.6921 mm/year. From the statistical data, the corrosion rate of the composites investigated can be formed in a descending order as: 0.3Zr<0.5Zr<0.7Zr. The polarization resistance of different weight percentages of zirconium can be concluded to be 0.3Zr = 29.426 Ω, 0.5Zr = 43.772Ω and 0.7Zr = 58.118Ω. From the above statistical data the polarization resistance of the composites investigated can be formed in a descending order as: 0.7Zr<0.5Zr<0.3Zr.

The above statements conclude that with increasing polarization resistance there is decrease in corrosion rate in the specimens and the polarization resistance increased with increase in the weight percentages of the zirconium in the aluminum.

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